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Synchrotron VUV and Soft X-Ray Radiation Effects on Aluminized Teflon® FEP

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SYNCHROTRON VUV AND SOFT X-RAY RADIATION EFFECTS ON ALUMINIZED TEFLON® FEP

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ABSTRACT

Surfaces of the aluminized Teflon® FEP multi-layer thermal insulation on the Hubble Space Telescope (HST) were found to be cracked and curled in some areas at the time of the second servicing mission in February 1997, 6.8 years after HST was deployed in low Earth orbit (LEO). As part of a test program to assess environmental conditions which would produce embrittlement sufficient to cause cracking of Teflon® on HST, samples of Teflon® FEP with a backside layer of vapor deposited aluminum were exposed to vacuum ultraviolet (VUV) and soft x-ray radiation of various energies using facilities at the National Synchrotron Light Source, Brookhaven National Laboratory. Samples were exposed to synchrotron radiation of narrow energy bands centered on energies between 69 eV and 1900 eV. Samples were analyzed for ultimate tensile strength and elongation. Results will be compared to those of aluminized Teflon® FEP retrieved from HST after 3.6 years and 6.8 years on orbit and will be referenced to estimated HST mission doses of VUV and soft x-ray radiation.

1. INTRODUCTION

Since the Hubble Space Telescope (HST) was deployed in low Earth orbit (LEO) in April 1990, two servicing missions have been conducted to upgrade its scientific capabilities. The first servicing mission (SM1) was conducted in December 1993, 3.6 years after deployment. The second servicing mission (SM2) was conducted in February 1997, 6.8 years after deployment. The HST servicing missions provided an opportunity for on-orbit examination and retrieval of second-surface metalized Teflon® FEP (fluorinated ethylene propylene) used as the top layer of multi-layer insulation (MLI) blankets and on radiator surfaces. Minor cracking of FEP surfaces on HST was first observed upon close examination of samples with high solar exposure retrieved during SM1 (1). During SM2, astronaut observations and photographic documentation revealed cracks in the FEP layer of the MLI on both solar-facing and anti-solar facing surfaces of the telescope.

The efforts reported in this paper were conducted as part of a test program to identify the LEO environmental constituent(s) responsible for cracking and embrittlement of Teflon® FEP on HST (2). Soft x-ray radiation from solar flares has been investigated previously as a possible cause for mechanical properties degradation of Teflon® FEP (3). This paper describes an investigation of the effects of vacuum ultraviolet (VUV) and soft x-ray radiation on Teflon® FEP. Samples of aluminized Teflon® FEP were exposed to synchrotron radiation of various VUV and soft x-ray wavelengths between 18 nm (69 eV) and 0.65 nm (1900 eV), and doses and fluences were compared to those estimated for the HST mission. Synchrotron radiation exposures were conducted using the National Synchrotron Light Source (NSLS), Brookhaven National Laboratory (BNL). Tensile testing was conducted on synchrotron radiation-exposed samples to determine tensile strength and elongation.

2. X-RAY AND SOLAR EXPOSURE ENVIRONMENT ON HST

Table 1 provides information about the x-ray and solar exposure environment on HST from deployment to SM1, SM2 and end-of-life (EOL) (4-6). Radiation in the x-ray wavelength regions 0.05-0.4 nm (3-25 keV) and 0.1-0.8 nm (1.5-12 keV) is attributed primarily to solar flares (3). Based on the x-ray fluence data for these two wavelength regions given in Table 1, it is evident that the majority of the solar flare x-ray fluence is in the 0.4 - 0.8 nm wavelength range, or 1.5-3 keV. The data in Table 1 are for the incident x-ray fluence and do not necessarily indicate the radiation dose absorbed by the Teflon® material.

TABLE 1. X-RAY AND SOLAR EXPOSURE ENVIRONMENT ON HST (4-6)

	SM1 3.6 years	SM2 6.8 years	EOL 20 years
X-ray fluence, 0.05-0.4 nm or 3-25 keV (J/m^2)	14.7	16	47.15
X-ray fluence, 0.1-0.8 nm or 1.5-12 keV (J/m^2)	222.6	252.4	699.6
Solar exposure for solar-facing surfaces (equivalent sun hours)	16,670	33,638	100,000

In addition to the data shown in Table 1 for the two regions of x-ray radiation, Reference 7 provides spectral flux data for VUV to soft x-ray wavelengths. From these data, HST fluences for narrow wavelength bands centered on wavelengths between 18 nm (69 eV) and 1.77 nm (700 eV), which were included in the synchrotron exposure experiments, were calculated and are shown in Table 2. Data for the additional synchrotron exposure energies up to 1900 eV were not available from this reference.

TABLE 2. SOFT X-RAY AND VUV FLUENCES FOR NARROW ENERGY BANDS ON HST

			Estimated Fluence on HST, Assuming Moderate Solar Activity (J/m^2)		
Energy Range, Ave. (eV)	Wavelength Range, Ave. (nm)	Moderate Solar Activity Flux ($photons/cm^2s$) (7)	SM1 (3.6 yrs.)	SM2 (6.8 yrs.)	EOL (20 yrs.)
68.6-69.4, 69	17.9-18.1, 18.0	1.10E+08	515	969	2943
283-297, 290	4.18-4.38, 4.28	4.00E+06	126	238	722
490-532, 510	2.33-2.53, 2.43	2.00E+06	133	251	761
663-742, 700	1.67-1.87, 1.77	1.00E+06	65	123	373

In order to determine the radiation dose absorbed by the Teflon® layer of the MLI, it is necessary to know how radiation is absorbed as a function of energy in Teflon®.

3. RADIATION ABSORPTION IN TEFLON®

Figure 1 shows the attenuation length of Teflon®, modeled as C_2F_4, as a function of energy (8). Attenuation length is defined as the depth into the material where the radiation intensity has fallen to 1/*e* (0.368) of its intensity at the surface. As shown, attenuation length is dependent on energy or wavelength. The absorption edges at approximately 290 and 700 eV are for carbon and fluorine, respectively. Crosses shown on the curve indicate the energies used for exposure of samples to synchrotron radiation, 69-1900 eV.

In order to calculate the absorbed radiation dose in Teflon®, it was necessary to use the fluence data for 0.05-0.4 nm and 0.1-0.8 nm along with the data for attenuation length. The equation for photon intensity at a specific energy as a function of depth in a material, I_E , is given as follows:

$$I_E = I_{0E}\, exp(-(\mu/\rho)x) \qquad [1]$$

where I_{0E} is the incident intensity at a specific energy, μ is the extinction coefficient, ρ is the density, and x is the depth into the material. The term μ/ρ is referred to as the linear absorption coefficient.

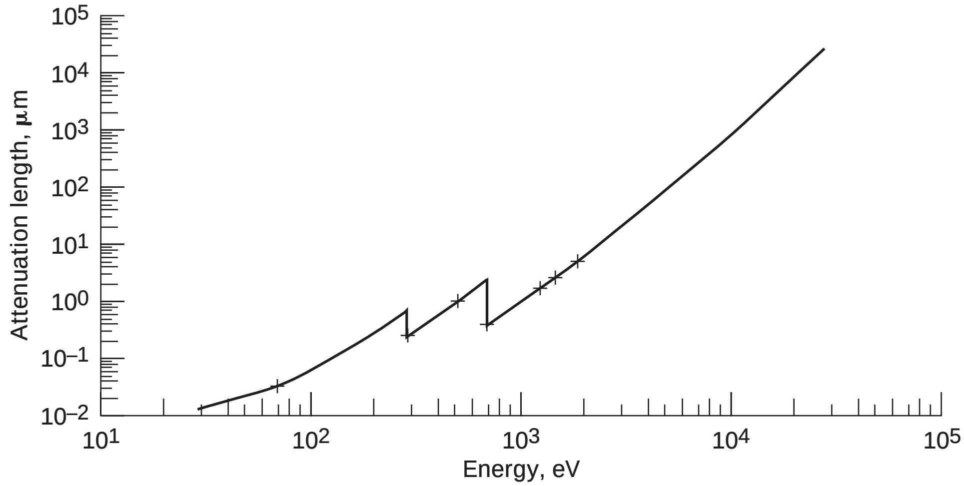

Figure 1.—Attenuation length of Teflon®, modeled as C_2F_4 (8).

The definition of attenuation length can be stated as the value of x where

$$I_E = I_{0E}/e. \qquad [2]$$

Substituting Equation 2 into Equation 1 gives:

$$I_{0E}/e = I_{0E}\, exp(-(\mu/\rho)x). \qquad [3]$$

Solving Equation 3 for μ/ρ gives:

$$1/x = \mu/\rho \qquad [4]$$

or

$$\text{attenuation length} = \rho/\mu.$$

Figure 1 provided x vs. energy, so, $1/x$ vs. energy can be obtained from the data in Figure 1. At energies above 1.5 keV, the energies provided for the HST exposure environment, the $1/x$ vs. energy data was curve-fitted to provide the linear absorption coefficient, μ/ρ, as a function of energy, E:

$$\mu/\rho = 3.46\text{x}10^9\, E^{-3.12}. \qquad [5]$$

Then, this was substituted into Equation 1 to give:

$$I_E = I_{0E}\, exp((-3.46\text{x}10^9\, E^{-3.12})x). \qquad [6]$$

Equation 6 provides the intensity of radiation at depth x into the material for a specific energy, I_E. Therefore, the absorbed intensity at a specific energy, A_E, is given as

$$A_E = I_{0E} - I_E \qquad [7]$$

or

$$A_E = I_{0E}\, (1 - exp((-3.46\text{x}10^9\, E^{-3.12})x)). \qquad [8]$$

Because I_{0E} and I_E represent intensity, they are expressed in units of W/m^2 or J/m^{2}s. Therefore, they can be described as values of x-ray flux at a specific wavelength or energy. A_E is then the absorbed x-ray flux at a particular energy. The incident HST x-ray fluence values indicated in Table 1 are reported in units of J/m^2 over a range of energies to indicate flux integrated over a specific mission duration. This can be represented by the following equation:

$$F_0 = \int I_{0t}\, dt \qquad [9]$$

where F_0 is the incident fluence over a specific mission duration given in J/m^2, I_{0t} is the incident flux at a specific time, and t is time. Multiplying both sides of Equation 8 by time, we obtain the following:

$$F_{AE} = F_{0E}\, (1 - exp((-3.46\text{x}10^9\, E^{-3.12})x)) \qquad [10]$$

where F_{AE} and F_{0E} are the absorbed and incident values of fluence, respectively, at a specific energy. Because fluence values in Table 1 are for the rather broad energy/wavelength bands indicated rather than for discreet energies/wavelengths,

$$F_{0E} = \frac{F_{0,E_1-E_2}}{E_2 - E_1}. \qquad [11]$$

Equation 11 was then substituted into Equation 10 and the result was numerically integrated over each energy range, 1.5-12 keV and 3-25 keV, using the HST incident fluence data from Table 1. For example, the numerical integration approximated the absorbed fluence for the 1.5-12 keV range given by the following equation:

$$F_{A,1.5\text{-}12\,keV} = \frac{F_{0,1.5-12keV}}{12-1.5} \int_{1.5keV}^{12\,keV} (1 - exp((-3.46\text{x}10^9\, E^{-3.12})x))\, dE. \qquad [12]$$

The absorbed x-ray radiation fluence was thus determined for specific depths, x, into the Teflon® material. Fluence of absorbed x-ray radiation was converted to dose, in units of rads, using the following equation:

$$\text{Dose, rads} = F_A (1/\rho)\ (1/x)\ (1\ \text{rad}/(0.01\ \text{J/kg})) \qquad [13]$$

where absorbed fluence over a particular energy range is F_A, in units of J/m^2; density, ρ, is in units of kg/m^3; and thickness, x, is in units of m. Because the outer layer of MLI on HST is comprised of 127 µm thick Teflon® FEP, absorbed radiation dose was calculated for this thickness and is shown in Table 3.

TABLE 3. X-RAY RADIATION DOSE ON HST FOR 127 µm FEP

	SM1 3.6 years	SM2 6.8 years	EOL 20 years
X-ray dose, 0.05-0.4 nm or 3-25 keV (krads)	0.99	1.08	3.18
X-ray dose, 0.1-0.8 nm or 1.5-12 keV (krads)	39.2	44.4	123

4. SYNCHROTRON RADIATION EXPOSURE CONDITIONS

Samples of second-surface aluminized Teflon® FEP were exposed to synchrotron radiation of various energies between 69 eV and 1900 eV. Two different beamlines at the NSLS were used to conduct experiments. As indicated in Table 4, Beamline U16B was used to provide radiation between 69 eV and 700 eV, and Beamline X8A was used to provide radiation between 510 eV and 1900 eV. Table 4 shows the energies and wave lengths used in these experiments including the calculated bandwidth at full-width, half-maximum and the estimated spot size for the beam. Also shown are attenuation lengths for each energy, as shown in Figure 1. As indicated by the attenuation lengths, the majority of radiation from these synchrotron radiation exposures is deposited within the first few micrometers of material.

TABLE 4. SYNCHROTRON RADIATION EXPOSURE CONDITIONS

NSLS Beamline Name	Energy (eV)	Wavelength (nm)	Approx. Spot Size, h x w (mm)	Attenuation Length, μm
U16B	69 ± 0.1	18.00 ± 0.026	3 x 5	0.033
	290 ± 1.7	4.28 ± 0.025	3 x 4.5	0.262
	700 ± 10	1.77 ± 0.025	3 x 4	0.403
X8A	510 ± 2.6	2.44 ± 0.012	10 x 4	1.06
	700 ± 3.5	1.77 ± 0.0089	10 x 4	0.403
	1256 ± 6.3	0.99 ± 0.005	10 x 4.5	1.74
	1489 ± 7.4	0.83 ± 0.004	10 x 4	2.74
	1900 ± 9.5	0.65 ± 0.003	10 x 4	5.36

5. EXPERIMENTAL PROCEDURES

5.1 Samples Samples for synchrotron radiation exposure were fabricated from Teflon® FEP of 127 μm thickness with approximately 100 nm of vapor deposited aluminum. Samples were exposed to the synchrotron radiation beam such that the FEP surface faced the beam and the aluminum surface was the back surface of each sample. Samples for tensile testing were “dog bone” shaped and were die-cut with a die manufactured according to ASTM Standard D638-95, Type V (9). For this sample size, the width of the narrow section of the dog bone was 3.18 mm, and the gauge length was 9.53 mm. The beam size for each synchrotron energy used is shown in Table 4 for comparison. For experiments at both beamlines, the beam covered the width of the narrow section of the dogbone. For experiments conducted at Beamline X8A where the beam was approximately 10 mm in height, the full gauge length of each specimen was illuminated by the beam. For samples exposed at Beamline U16B, the full gauge length was not covered by the beam. Additional samples included in the experiments which were not intended for tensile testing were nominally 3 to 5 mm maximum width and 15 mm length.

5.2 Apparatus Figure 2 shows the sample/detector holder which was able to hold up to seven pairs of samples as shown. When pairs of samples were exposed, each pair consisted of one tensile specimen and one non-tensile specimen.

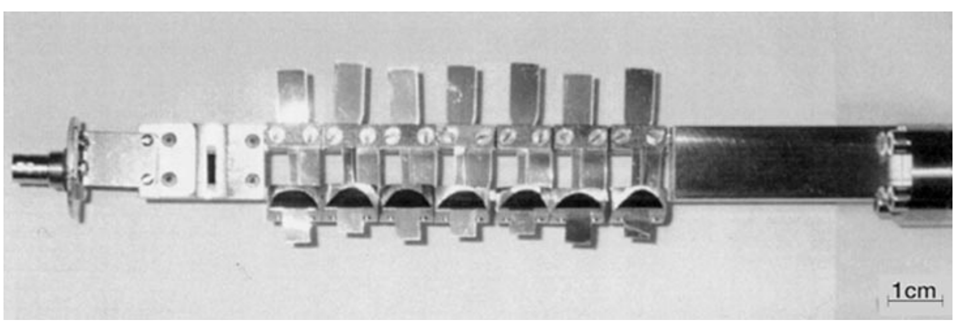

Figure 2.—Sample holder for synchrotron radiation exposure of FEP samples.

In most cases, the tensile specimen was centered on the synchrotron beam, and the other sample received the “tail” of the synchrotron beam, typically a small fraction of the intensity provided by the beam center. Shown at the left of the sample holder is the silicon photodiode used to measure the synchrotron beam intensity for each sample exposure. The sample/detector holder was attached to a linear positioner with a movement scale allowing positioning accurate to ± 0.01 mm. Prior to installation in the vacuum chamber, distances between the photodiode and samples were measured with digital calipers to assure accuracy in placement of the samples in the synchrotron beam. Measurements of synchrotron beam intensity were made before and after each exposure and were averaged to determine photon fluence for each sample exposed. A mask was used over the photodiode providing a measurement area 3 mm wide by 10 mm high, comparable to the gauge size of a tensile specimen.

5.3 Tensile Testing Tensile testing was conducted to determine degradation in tensile strength and elongation for synchrotron radiation-exposed FEP samples as compared to pristine material. Sample dimensions were described in Section 5.1. Samples exposed at Beamline X8A were tensile tested using an Instron 4505 load frame with Instron Series IX data acquisition software. Samples exposed at Beamline U16B were tensile tested using an automated bench-top tensile tester and data acquisition software. A strain rate of 0.212 mm/s was used for all tests. Because samples from each beamline were tested using different instruments at different times, results for each set of synchrotron-exposed samples were compared to a set of pristine samples tensile tested at the same time.

6. RESULTS AND DISCUSSION

Table 5 shows results of tensile testing of synchrotron radiation-exposed second surface aluminized FEP. For each sample the exposure energy, measured fluence, absorbed dose, and comparison to orbital doses are provided. Samples U5T and U2T were exposed to both 290 eV and 700 eV radiation, sequentially, as shown. The objective of these experiments was to determine whether exposure to radiation at both the carbon and fluorine absorption edges would produce synergistic degradation.

The HST EOL 0.05-0.4 nm (1.5-25 keV) doses at the attenuation length of the exposure energy were calculated using the data in Table 1 and equations 1-11. For 69-700 eV the narrow-band energy doses for HST EOL at the attenuation length of the exposure energy are also shown for comparison. These narrow-band energy dose values were calculated using the data in Table 2 and equations 2, 7, 9, and 13. As shown in Table 5, in most cases, the sample dose significantly exceeded the HST EOL dose.

The ultimate tensile strength (UTS) and elongation were measured for each tensile specimen, and data are provided in Table 5 with the percent changes from pristine specimens. The tensile test data for pristine specimens are given in Table 6. The tensile test data in Table 5 can be compared to the changes in tensile strength and elongation experienced for aluminized FEP materials retrieved from HST during the first and second servicing missions which are provided in Table 7 (10).

For most synchrotron radiation-exposed samples, some degradation in tensile strength and elongation was observed. However, the degradation in tensile strength and elongation experienced by the synchrotron radiation-exposed samples was not comparable to that demonstrated by samples retrieved from HST after 3.6 years and 6.8 years. The worst degradation in tensile strength was 30.3%, for sample X1T, which received 256 times the HST EOL x-ray dose or an equivalent of over 5000 years in space. This result can be compared to that of an HST specimen retrieved during SM1 after 3.6 years in space. This material showed a degradation in tensile strength of 37-42%, greater than that of the worst damaged synchrotron-exposed sample. The synchrotron radiation-exposed sample which experienced the greatest degradation in elongation was X14T which showed a degradation in elongation of 83%. This sample received an equivalent of over 1500 times the HST EOL x-ray dose or an equivalent of over 30,000 years in space. Compare this result to the complete loss of elongation for the material retrieved from HST after 6.8 years. It is obvious from these results that at the energies used for these synchrotron radiation exposures, VUV radiation and soft x-ray radiation were not sufficient to cause the degradation in mechanical properties displayed by 6.8 year HST-exposed aluminized FEP.

All synchrotron-exposed tensile specimens were examined using optical microscopy for evidence of surface embrittlement or cracking. This effect was described in Reference 3. The only specimens showing evidence of surface cracking after tensile testing were those samples exposed to 290 eV synchrotron radiation, samples U1T and U4T. Figure 3 shows a photomicrograph of sample U4T after tensile testing as compared to sample U6T, exposed to 69 eV synchrotron radiation of a higher absorbed radiation dose. Sample U6T was comparable to all other synchrotron-exposed specimens in that it showed no evidence of surface cracking. It is possible that because 290 eV is at the carbon absorption edge, more severe degradation in the radiation-absorbing layer may occur.

TABLE 5. TENSILE TESTING OF SYNCHROTRON RADIATION-EXPOSED FEP SAMPLES

Sample Label	Energy (eV)	Incident Fluence (J/m^2)	Dose at atten. length (krads): Sample Dose	Dose at atten. length (krads): HST EOL 0.05-0.8 nm Dose	Dose at atten. length (krads): HST EOL Narrow Energy Band Dose	Ultimate Tensile Strength, UTS (MPa)	Elongation (%) at Break	% Change in UTS from pristine	% Change in % Elongation from pristine
U3T	69	5304	4.73E+06	839	2.7E+06	19.48	214	0.2	4.4
U6T	69	5076	4.52E+06			17.29	176	-11.0	-14.1
U4T	290	3136	3.52E+05	825	5.6E+04	15.08	107	-22.4	-47.8
U1T	290	3099	3.48E+05			14.76	114	-24.1	-44.4
U5T	290	1583	1.78E+05	825	5.6E+04	> 14.34*	> 134*	> -26.2*	> -34.6*
	700	1534	1.12E+05	816	2.1E+04				
U2T	290	1611	1.81E+05	825	5.6E+04	14.25	87.6	-26.7	-57.3
	700	1521	1.11E+05	816	2.1E+04				
X6T	510	474	1.31E+04	777	1.6E+04	22.36	649	16.0	11.1
X17T	700	973	7.10E+04	816	2.1E+04	16.18	434	-16.1	-25.7
X15T	700	3055	2.23E+05			17.60	489	-8.7	-16.3
X16T	700	10347	7.55E+05			16.97	484	-12.0	-17.2
X10T	1256	17982	3.04E+05	742	N/A	16.23	406	-15.8	-30.4
X3T	1489	161	1.73E+03	695	N/A	20.34	626	5.5	7.2
X2T	1489	8211	8.81E+04			20.56	629	6.7	7.7
X1T	1489	16123	1.73E+05			13.45	194	-30.3	-66.8
X8T	1489	21938	2.35E+05			14.36	322	-25.5	-44.8
X14T	1489	95859	1.03E+06			13.85	98	-28.2	-83.2
X4T	1900	32	1.76E+02	599	N/A	17.30	516	-10.3	-11.6
X5T	1900	3014	1.65E+04			19.32	596	0.2	2.0
X12T	1900	6632	3.64E+04			16.58	440	-14.0	-24.6

*Due to power failure, test stopped at this point prior to sample break

TABLE 6. TENSILE TESTING OF PRISTINE ALUMINIZED FEP

Specimen Description	Ultimate Tensile Strength (MPa)	Elongation (%)
X7T, Unexposed witness	20.48	600.3
Pristine, Avg. for 5 specimens, comparison for U samples	19.43 ± 1.9	205 ± 28
Pristine, Avg. for 3 specimens, comparison for X samples	19.28 ± 3.2	584 ± 126

TABLE 7. TENSILE TEST DATA OF HST-EXPOSED MATERIALS (10)

Sample	Yield Strength (MPa)	Ultimate Tensile Strength, UTS (Mpa)	% Change in UTS from Pristine	Elongation (%)	% Change in % Elongation from Pristine
Pristine aluminized FEP, 3 samples	14.1±0.3	26.5±1.7		363±25	
SM1-retrieved MLI, 3.6 yrs., 11,339 ESH	14.3 14.3	15.4 16.6	-42 -37	196 116	-46 -68
SM2-retrieved MLI, 6.8 yrs., 33,638 ESH	N/A N/A	13.2 2.2	-50 -92	0 0	-100 -100

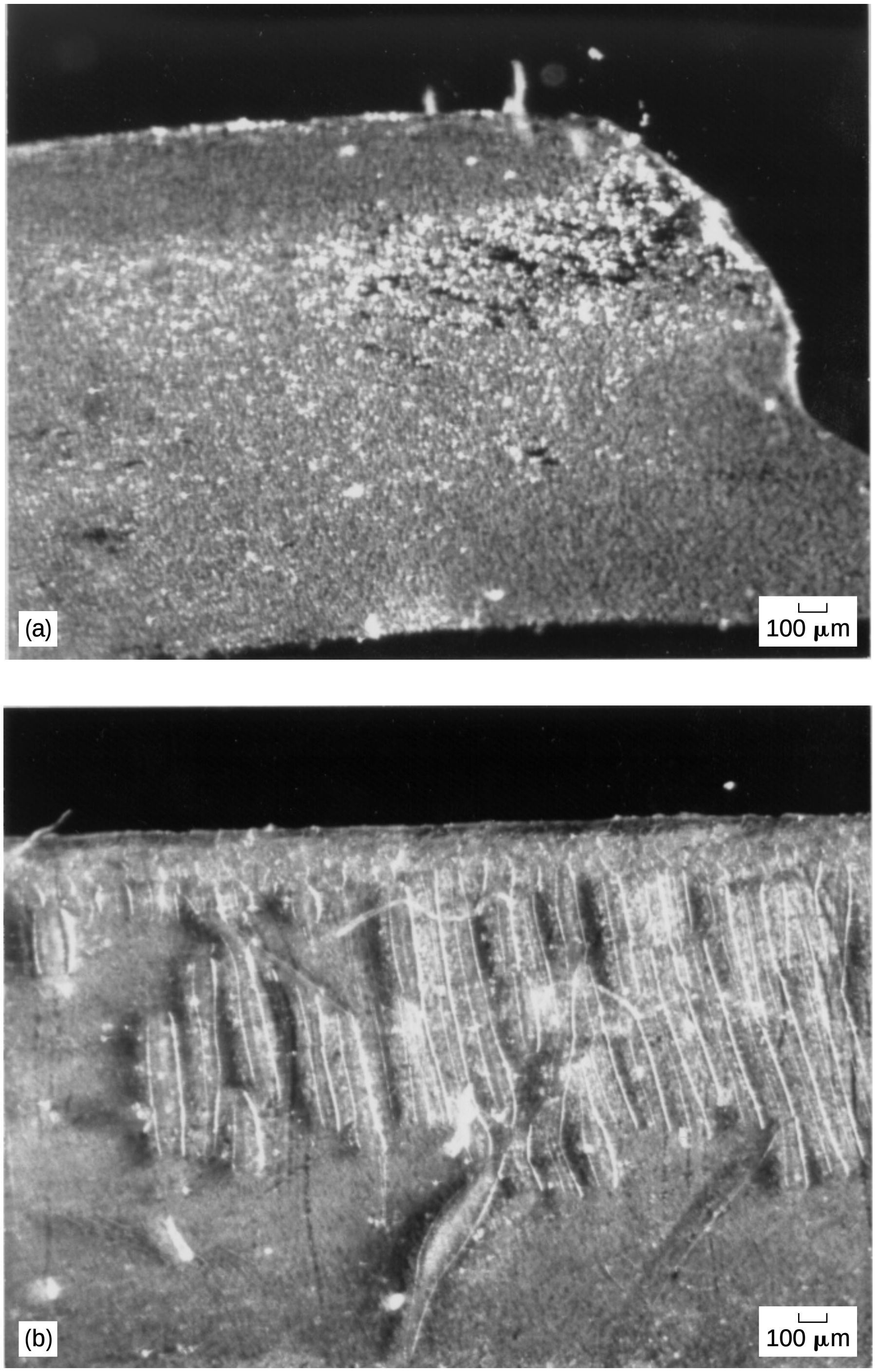

Figure 3.—Photomicrographs of tensile-tested, synchrotron radiation-exposed samples. (a) U6T exposed to 5076 J/m^2 at 69 eV. (b) U4T exposed to 3136 J/m^2 at 290 eV.

7. CONCLUSIONS

Exposure to synchrotron radiation in the VUV and soft x-ray range of energies is capable of causing degradation in the mechanical properties of Teflon® FEP. However, FEP samples exposed to synchrotron radiation of doses significantly greater than HST EOL doses did not show loss of tensile strength or elongation comparable to that of the severely embrittled aluminized FEP retrieved from HST during SM2 which experienced 6.8 years on orbit. Some evidence of wavelength-dependence of damage was observed for samples exposed to 290 eV synchrotron radiation which is at the carbon absorption edge. These samples showed surface cracking after tensile testing. Based on these results, exposure to VUV and soft x-ray radiation alone is not sufficient to cause the severe degradation in mechanical properties observed for FEP materials exposed to the HST environment.

8. ACKNOWLEDGMENTS

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13. ABSTRACT ***(Maximum 200 words)***

Surfaces of the aluminized Teflon® FEP multi-layer thermal insulation on the Hubble Space Telescope (HST) were found to be cracked and curled in some areas at the time of the second servicing mission in February 1997, 6.8 years after HST was deployed in low Earth orbit (LEO). As part of a test program to assess environmental conditions which would produce embrittlement sufficient to cause cracking of Teflon® on HST, samples of Teflon® FEP with a backside layer of vapor deposited aluminum were exposed to vacuum ultraviolet (VUV) and soft x-ray radiation of various energies using facilities at the National Synchrotron Light Source, Brookhaven National Laboratory. Samples were exposed to synchrotron radiation of narrow energy bands centered on energies between 69 eV and 1900 eV. Samples were analyzed for ultimate tensile strength and elongation. Results will be compared to those of aluminized Teflon® FEP retrieved from HST after 3.6 years and 6.8 years on orbit and will be referenced to estimated HST mission doses of VUV and soft x-ray radiation.

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